

Igniter Induced Hybrids in the 20-L Sphere

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Abstract

The explosibility characteristics of a combustible dust are traditionally described by two parameters, the maximum explosion pressure P_{\max} and the deflagration index K_{St} . P_{\max} and K_{St} are determined through testing in a closed pressure resistant spherical vessel, usually a 20-L sphere, and constitute key variables in the design of explosion protection systems, such as venting, suppression or isolation systems.

The potential for overdriving dust combustion with pyrotechnical igniters in the 20-L sphere has been recognized and discussed for many years, notably in the determination of the minimum explosible (MEC) and limiting oxygen concentrations (LOC), which has led to specific guidelines regarding the ignition source strength in ASTM standards.

The present paper presents experimental evidence that pyrotechnical igniters may, in some instances, physically alter the dust being tested in a 20-L sphere in such a way, that a turbulent gas and dust hybrid mixture is formed. In these cases, it is shown that K_{St} values obtained in the 20-L sphere may be several times greater than in the 1-m³ chamber. Therefore, the results from 20-L sphere testing are no longer representative of a dust deflagration in a real process environment. For these samples, testing in a 1-m³ chamber may be more appropriate to determine the explosibility parameters, especially the deflagration index, and thus the design of the explosion protection system.

Keywords: dust, deflagration, igniter, overdriving, hybrid

1. Introduction

A dust explosion occurs when an airborne combustible dust cloud encounters an effective ignition source. The resulting pressure (and temperature) increase can severely injure people and damage surrounding equipment and buildings, and therefore needs to be controlled.

The severity of a dust explosion is described by two parameters P_{\max} and K_{St} , respectively the maximum explosion pressure and the deflagration index, which is the product of the maximum rate of pressure rise and the cube root of the vessel volume.

K_{St} and P_{\max} are determined through testing in a closed pressure resistant spherical vessel: a dust cloud of fixed concentration is dispersed in the vessel and ignited at its center by pyrotechnical igniter(s). P_{\max} is determined based on the maximum pressure reached during the deflagration test, while K_{St} is calculated using the slope of the steepest part of the pressure-versus-time curve recorded during the deflagration.

A 20-L sphere apparatus, as well as a modified testing protocol, has been developed by Siwek (1977) as an alternative for the 1-m³ chamber introduced by Bartknecht (1981) in the early 1970's to propose cheaper and faster tests. While several modifications (volume of the dust container, ignition delay time, dispersion systems) were made so the results found in the 20-L sphere would match the results of the 1-m³ chamber (Figure 1), the same pyrotechnical igniters (10-kJ) were kept to perform explosion tests in the 20-L sphere.



Figures 1: Pictures of 20-L sphere and 1-m³ chamber operated by Fike Corporation

The current paper presents experimental evidence that the strong pyrotechnical igniters employed for dust explosibility testing may, in some instances, physically alter the dust being tested in a 20-L sphere in such a way, that a hybrid mixture is formed. It is likely that the elevated temperature from the igniters affects the dust being tested prior to the actual arrival of the flame front, and that this elevated temperature is responsible for the physical changes in the dust that result in a turbulent gas and dust mixture. This behavior may depend on the physical properties of the dust, i.e. its ability to generate combustible gases in a very short period of time upon heating. This may be described, in some instances, by the flash point or the devolatilization rate of the dust being tested. In these cases, it is shown that K_{St} values obtained in the 20-L sphere may be 2 to 4 times greater than in the 1-m³ chamber. Therefore, the results from 20-L sphere testing are no longer representative of a dust deflagration in a real process environment.

2. Effect of ignition energy on explosive properties: previous experimental investigations

2.1 Zhen and Leuckel (1997)

Zhen and Leuckel (1997) were among the firsts to recognize, describe and study the effects of pyrotechnic igniters on dust explosions. According to the authors, pyrotechnical igniters accelerate the burning rate during an explosion due to volumetric and/or multipoint ignition effect. The extent of this overdriving is related not only to the energy of the igniters, but also to the reactivity of the mixtures. The igniter effect is more important for the early stages of flame propagation, thus more significant in small explosion chambers.

Zhen and Leuckel conducted dust explosion tests in a 1-m³ chamber with cornstarch using 10-kJ and 75-J pyrotechnical igniters. Results are presented in Figure 2. While K_{St} values are different (in relative terms) at a dust concentration of 250 g/m³, these differences tend to be less for a dust concentration of 500 g/m³, i.e. when the reactivity (dP/dt_{max}) increases.

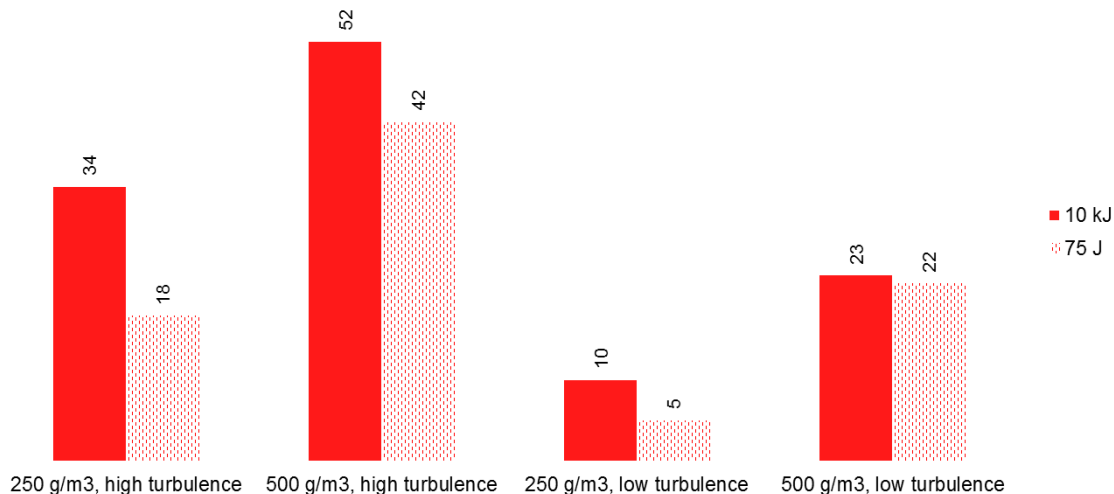


Figure 2: K_{St} values obtained for cornstarch in a 1-m³ vessel at different concentrations and turbulence intensities (Zhen and Leuckel, 1997)

Zhen and Leuckel also report vented explosion tests (Fields, 1982) showing that the explosion pressures developed from the guncotton ignition source were as much as five times higher than when a point spark source was used.

2.2 Going, Chatrathi and Cashdollar (2000)

Going, Chatrathi and Cashdollar (2000) present a comparison of minimum explosible concentration (MEC) and limiting oxygen concentration (LOC) determination in a 20-L sphere to a 1-m³ chamber.

All tested dusts exhibit comparable median particle size (between 20 and 44 μm) and have low moisture content (below 3%). On the other hand, they ~~widely~~ differ in terms of chemistry and volatiles content (Table 1).

Dust	Median particle size (μm)	Volatile Content (%)
Tetramethylpiperidine (RoRo93)	29	100
Pittsburgh coal	44	37
Gilsonite	28	84
Lycopodium	28	92
Aluminum	20	NA
Iron	23	NA

Table 1. Physical and chemical properties of dusts
(Going, ~~Chatrathi and Cashdollar~~, 2000)

MEC testing

Results of their investigation are summarized on Figure 3. They show that the measured MECs from the 1-m³ chamber are essentially independent of ignition energy over the range studied, which is not the case with the 20-L sphere: as the energy increased from 2.5-kJ, the apparent MEC decreased and was notably ~~less~~ than the 1-m³ chamber results for the carbonaceous dusts. The authors attributed this behavior to overdriving in the smaller vessel due ~~a too~~ strong ignition source.

For the carbonaceous dusts, the closest agreement to the 1-m³ chamber data was found with a 2.5-kJ ignitor in the 20-L sphere, except for the Pittsburgh coal dust, for which the best agreement is found with a 1-kJ ignition source. The data also show that the more difficult-to-ignite dusts (such as iron) with higher MEC values would require a higher ignition energy of 5-kJ.

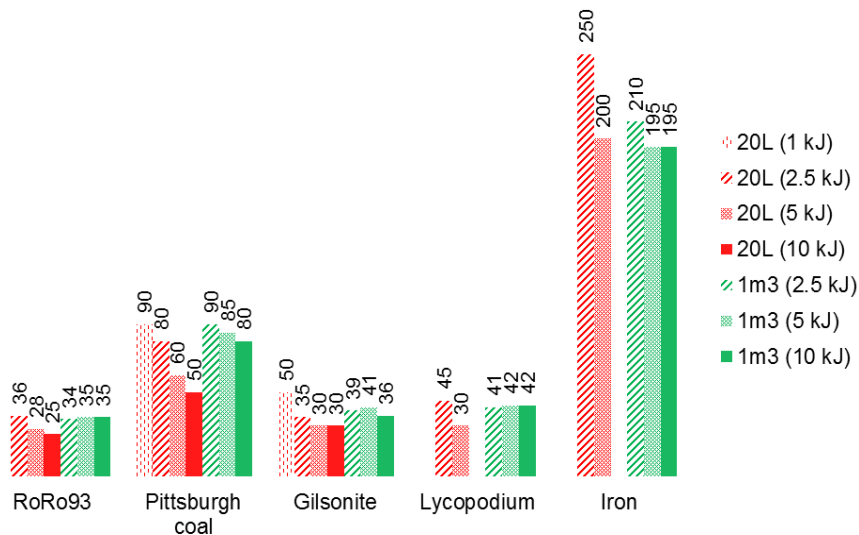


Figure 3. Results of MEC testing (g/m^3) for five dusts
(Going, Chatrathi and Cashdollar, 2000)

LOC testing

All of the LOC experiments in the 1-m³ chamber were conducted with the 10-kJ ignition source and the 20-L sphere tests were conducted mainly with 2.5-kJ or 5-kJ ignition sources (Figure 4).

Comparing the LOCs of aluminum and RoRo93 measured in the 20-L sphere with the LOCs measured in the 1-m³ chamber leads to the conclusion that 2.5-kJ is an appropriate ignition source for the 20-L sphere. However, the same 2.5-kJ ignition source does overdrive the gilsonite and Pittsburgh coal dusts, therefore a 1-kJ ignition source may be more suitable for these latter dusts in the 20-L sphere.

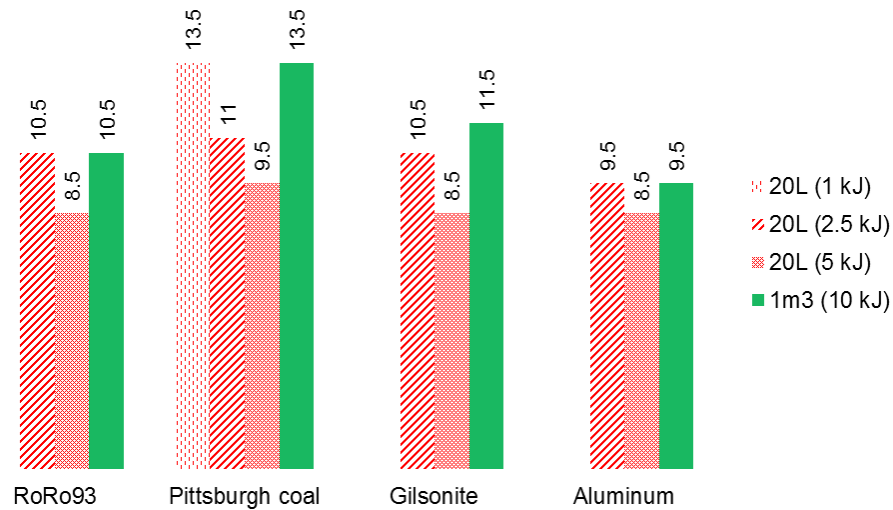


Figure 4. Results of LOC testing (% O₂) for four dusts (Going, Chatrathi and Cashdollar, 2000)

In both experiments, the authors showed that overdriving would occur with large ignition sources, and concluded that a 2.5-kJ ignition source in the 20-L sphere would yield approximately the same result as a 10-kJ source in the 1-m³ chamber. The authors recommended the 1-m³ chamber for measuring LOC values below 10%.

The use of 2.5-kJ pyrotechnical igniters is recommended in ASTM standards related to LOC (E2931) and MEC (E1515) determination (Table 2).

	Bureau of Mines 20-L Chamber		Fike 1 m ³ Chamber
	2.5-kJ	5-kJ	10-kJ
Bituminous coal, Pocahontas seam	120	85	...
Bituminous coal, Pittsburgh seam	80	60	80
Lycopodium	45	30	42
Gilsonite	35	30	36
Polyethylene	32	28	...

Table 2: Comparison of MECs determined in a 20-L chamber and 1-m³ chamber with different pyrotechnical igniters (ASTM E1515)

2.3 Dastidar and Amyotte (2002)

Dastidar and Amyotte (2002) compared minimum inerting concentration (MIC) measurements in the 20-L sphere and 1-m³ chamber. Initial experiments performed with a 5-kJ ignition source led to much greater MIC in the 20-L sphere than in the 1-m³ chamber. Using a 1-kJ igniter reduced MIC values, becoming closer to the 1-m³ chamber values.

Figure 5 shows the different minimum inerting concentrations envelopes obtained for Pittsburgh pulverized coal using monoammonium phosphate (MAP) as an inertant. Using a 5-kJ ignition source in the 20-L sphere largely overestimates the envelope determined in the 1-m³ chamber. Using lower energies gradually provided better agreement. The inerting level plotted on the graph for the 20-L sphere, using a 0.5-kJ igniter, ultimately overlaps data points from the 1-m³ chamber.

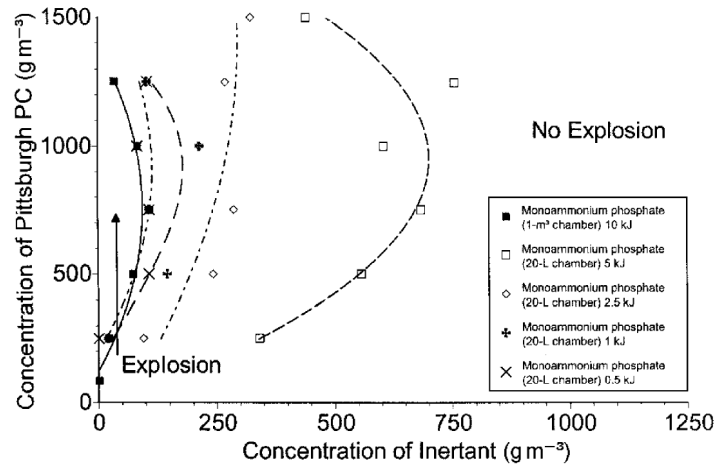


Figure 5: Comparison of 1-m³ and 20-L chamber inerting curves at different ignition energies for Pittsburgh pulverized coal using monoammonium phosphate as inertant (Dastidar and Amyotte, 2002)

The same agreement is seen for cornstarch using sodium bicarbonate as an inertant but at a different ignition strength of 1-kJ in the 20-L sphere (Figure 6).

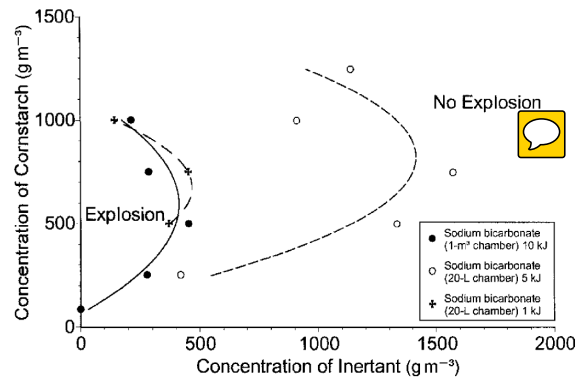


Figure 6: Comparison of 1-m³ and 20-L chamber inerting curves at different ignition energies for cornstarch using sodium bicarbonate as inertant (Dastidar and Amyotte, 2002)

Aluminum, on the other hand, did not need a reduction in the ignition energy to produce similar results in both chambers.

The authors conclude that the ignition energy required to produce inerting results in the 20-L chamber similar to a 1-m³ volume is dependent on the material being tested, see Figure 7.

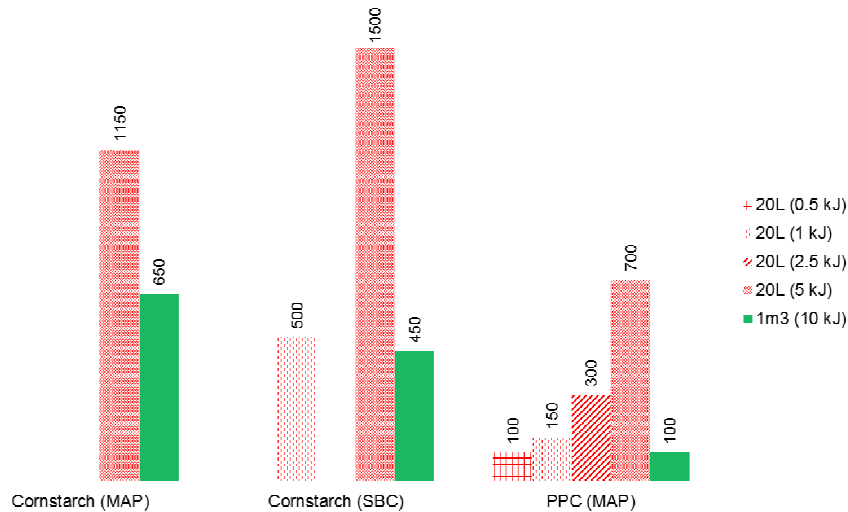


Figure 7: MIC values for cornstarch and PPC (Dastidar and Amyotte, 2002)

2.4 Proust et al. (2007)

Proust et al. (2007) performed a study of the measured K_{St} with both 20-L and 1-m³ test vessels using a 10-kJ ignition energy in both vessels. While the correlation in the results between the two vessels was reasonable, four of the dusts tested that had low K_{St} values in the 20-L vessel (sodium monochloroacetate, Lixivalt, Metco, and solid sewing residues) were found to be non-explosible when run in the 1-m³ vessel. Proust et al. suggested that a dust with a K_{St} of 45 bar.m/s as measured in the 20-L test would likely be shown to be non-explosible when tested in a 1-m³ vessel (Figure 8).

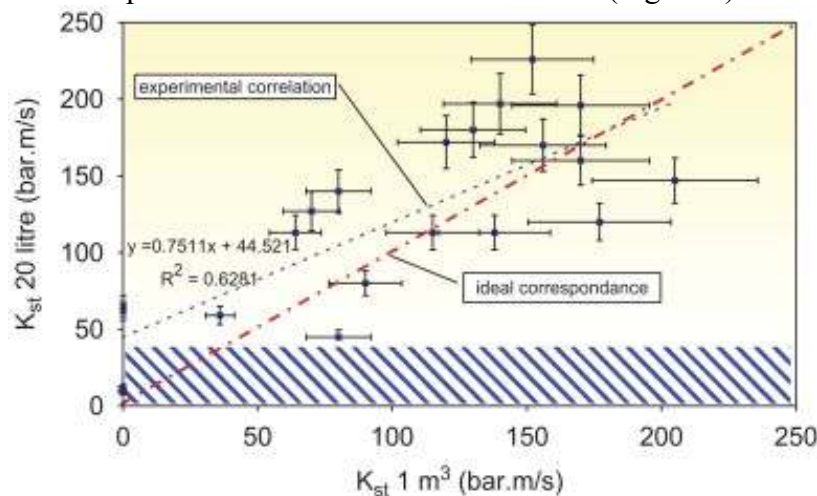


Figure 8: K_{St} obtained with the 20-L sphere and the 1-m³ vessel (Proust et al, 2007)

2.5 Thomas, Kirby and Going (2013)

Thomas, Kirby and Going (2013) conducted screening explosibility tests per ASTM E1226 with urea dust in both a 20-L sphere (with either 1 or 2 x 5kJ igniters) and 1-m³ chamber. They observed positive test in the small vessel, but negative test in the large vessel (Table 3). They conclude that the “false positive” result obtained in the 20L sphere was the result of overdriving the combustion reaction, while testing in the 1-m³ chamber allowed the urea dust to be properly characterized. They recommend testing low-K_{St} dusts in a vessel larger than a 20-L volume, where the flame must propagate over a more reasonable distance in order to develop a maximum explosion pressure P_{\max} value sufficient to classify the dust as explosible.

Vessel	Igniter	Result
20L	2 x 5-kJ	Ignition $P_{\max} = 5.4$ bar $K_{\max} = 21$ bar.m./s
20L	1 x 5-kJ	No ignition
1m ³	2 x 5-kJ	No ignition
1m ³	2 x 10-kJ	No ignition

Table 3. Results of screening tests with urea (Thomas, Kirby & Going, 2013)

2.6 Kuai et al. (2013)

Kuai et al. studied the effect of ignition strength (1-kJ, 2-kJ, 5-kJ and 10-kJ pyrotechnical igniters) on the maximum explosion pressure and the minimum explosible concentration (MEC) of sweet potato, magnesium and bituminous coal dusts in a 20-L sphere. They concluded that MEC is significantly affected by ignition energy (Figure 9), and suggested to use different ignition energies depending on the nature of the dust (5 to 7-kJ for carbonaceous dusts, 2 to 5-kJ for light metals), thus corroborating the earlier conclusions of Going et al..

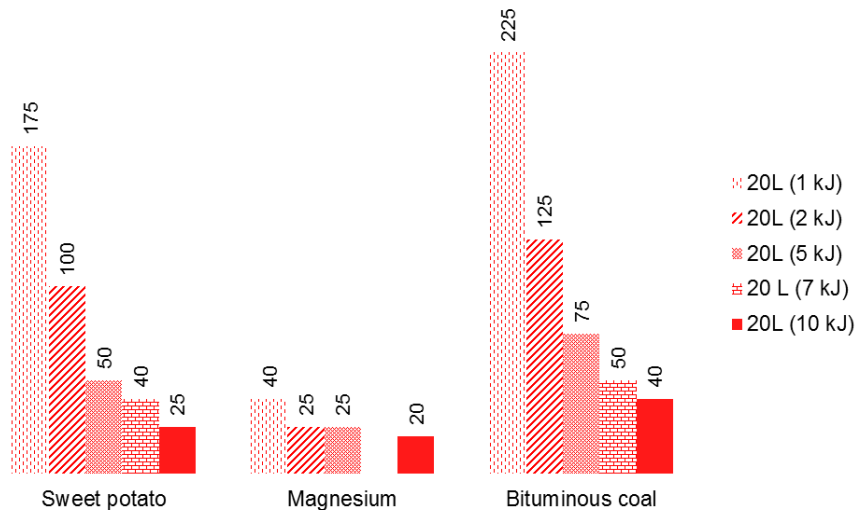


Figure 9: MEC values obtained for sweet potato, magnesium and bituminous coal in a 20-L sphere using different ignition strengths (Kuai et al., 2013)

3. Pyrotechnical igniters

Dust mixtures require a (much) higher energy than gaseous mixtures to be ignited. This may be up to several orders of magnitude, depending on the nature of the dust, its particle size, moisture content, and other factors.

While fuse wires are employed to ignite gaseous mixtures (several Joules), pyrotechnical igniters (several kilo-Joules) are used for dust testing (Figure 10). These powerful igniters are typically made of 40 % zirconium, 30 % barium nitrate and 30 % barium peroxide.



Figure 10: Pyrotechnical igniters (Sobbe)


Figure 11 shows a 5-kJ pyrotechnical igniter fired in an open 20-L sphere. The hot gas and particles fill the entire volume (potentially acting like a multiple ignition source). This is in contrast with sparks, or even fuse wires used for gas testing.



Figure 11: Visualization of the fireball and hot particles generated by a 5-kJ pyrotechnical igniter in a 20-L sphere

3.1 Pressure increase due to pyrotechnical igniters

Figure 12 compares the pressure developed by different pyrotechnical igniters as measured in a 20-L sphere and in a 1-m³ chamber by the present authors.

Note that Cashdollar and Chatrathi (1992) report a pressure rise of about 0.54 bar with a 5-kJ ignitor in the 20-L sphere, compared to 0.03 bar with a 10-kJ ignitor in a 1-m³ vessel, which agrees with the present authors' data. 

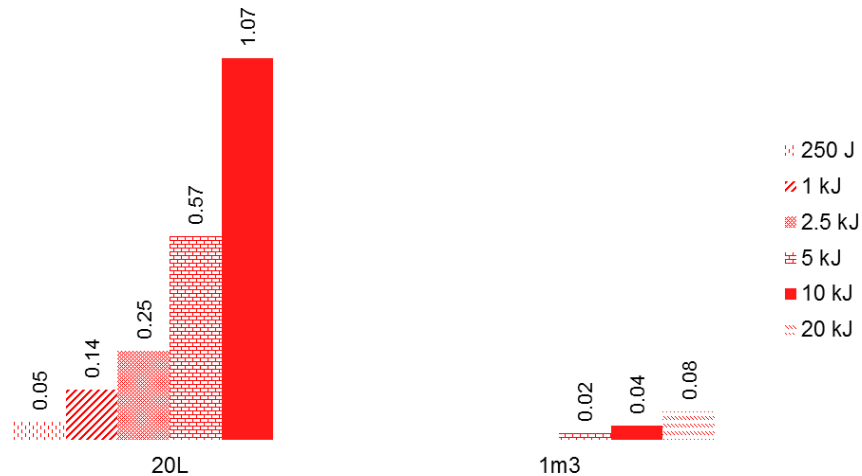


Figure 12: Pressure increase (in bar) due to pyrotechnical igniters in a 20-L sphere and 1-m³ vessel

One can see that the pressure increase is negligible in the 1m³ vessel. This is not the case in the 20-L sphere, with an increase of more than 1 bar when using 2 x 5-kJ igniters. It can be concluded that the pyrotechnical igniters significantly alter the initial pressure inside the 20-L sphere, while this effect is barely noticeable in a volume 50 times larger.

Figure 13 presents the equivalent pressure rate of rise normalized by the vessel volume (similar to K_{St} or K_G) of the pyrotechnical igniters used alone (no combustible dust present in the vessel).

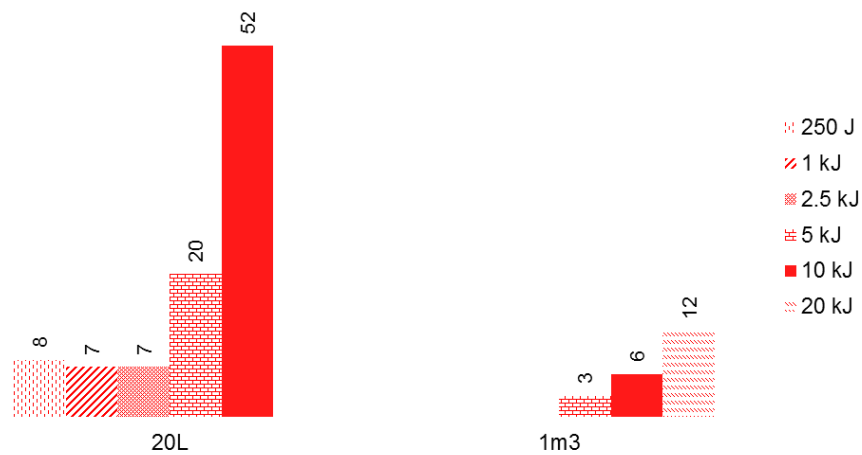


Figure 13: Pressure rate of rise due to pyrotechnical igniters in a 20-L sphere and 1-m³ vessel

An equivalent deflagration index of 50 bar.m/s is created with 2 x 5kJ igniters, which is significant.

3.2 Temperature increase due to pyrotechnical igniters

Based on the pressure rise developed in the 20-L sphere by one 5-kJ igniter (0.57 bar) and using the ideal gas law, the [macroscopic](#) temperature rise would be about +190°C. However, one can expect much higher temperatures locally.

Scheid et al. (2013) reported high-speed images for the firing of fuse wires and pyrotechnical igniters in an 11-L windowed autoclave. Using a fast IR camera, they were able to estimate the maximum temperature level in the generated flame/arc and the maximum volume of flame/arc.

Figure 14 shows four sequences of the flame/arc propagation of exploding wire (first and third sequence) and pyrotechnical igniter (second and fourth sequence) recorded with the IR camera. The sequences show a period of approx. 5 ms within the first 15 ms after triggering the igniter.

The temperature range in the first two sequences was adjusted such that temperatures between 200°C and 650°C can be observed, while temperatures between 650°C and 2,000°C can be visualized on rows three and four.

A comparison between sequence one and sequence two shows that the volume with the highest temperatures generated from the pyrotechnical igniter is much larger than the volume with such temperatures generated from the exploding wire. On sequence four, it can also be observed that the volume of gas having a temperature between 650°C and 2,000°C is still significant in the case of the pyrotechnical igniters.

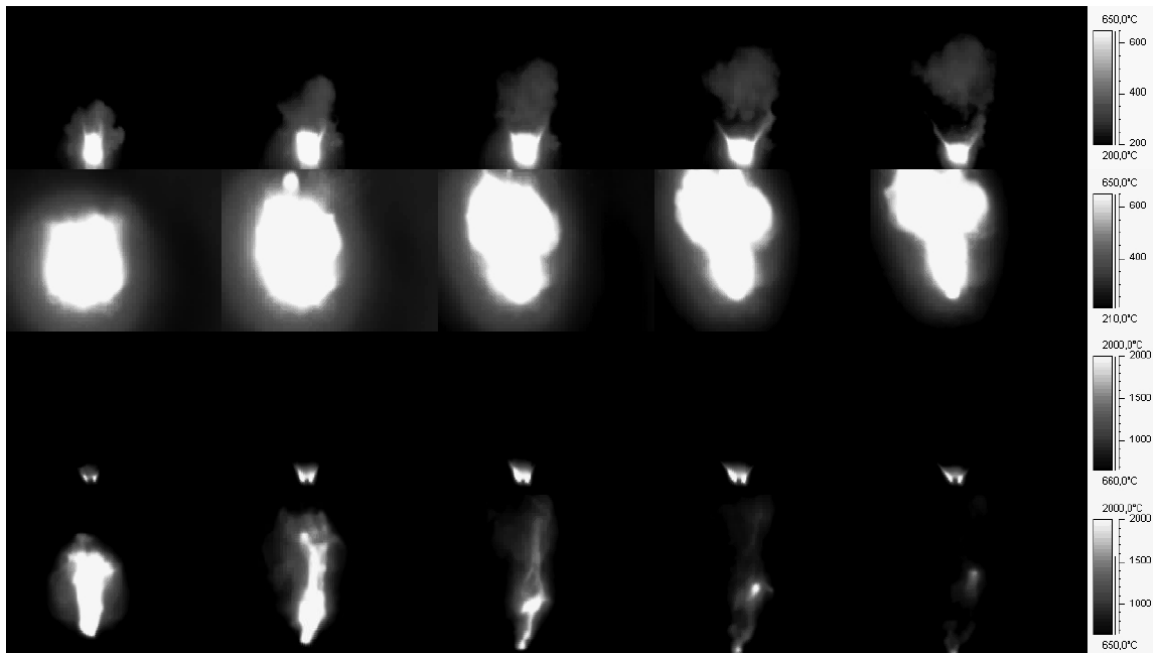


Figure 14: Visualization of the maximum flame/arc volume for an exploding wire (row 1 and 3) and a pyrotechnical igniter (row 2 and 4), from (Scheid et al, 2013)

It is seen that temperatures of 650°C or higher can be reached within a small volume when using pyrotechnical igniters. It is easy to hypothesize that such temperature rise can lead to a significant alteration of the sample, and this is prior to the arrival of the flame front. Depending on its physical behavior, a dust sample may partially decompose, forming or releasing a low boiling liquid or gas. As an example, a material like anthraquinone, discussed in the following paragraphs, has a flash point of only 185°C.

High-speed recordings displayed on Figure 15 show the progression of the flame induced by 2 x 5-kJ pyrotechnical igniters in an open 1-m³ chamber. The volume occupied by the fireball is not negligible but is far less than in the case of the 20-L sphere (Figure 11).

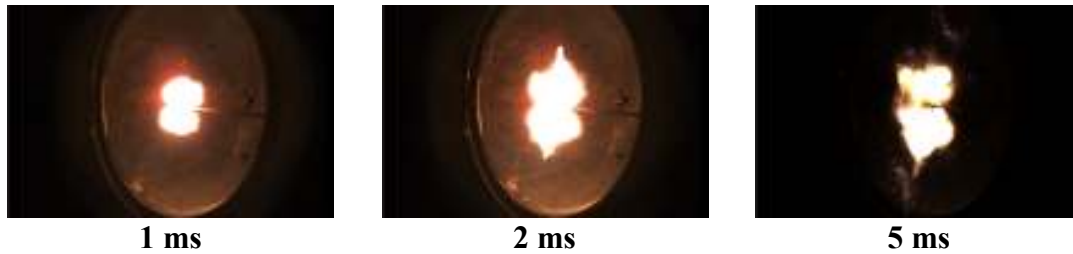


Figure 15: Visualization of the fireball generated by 2 x 5-kJ pyrotechnical igniter in a 1-m³ chamber

4. Effect of ignition energy on the explosibility parameters of some dusts

Hybrid deflagrations are understood to be the result of ignition of a turbulent gas-dust mixture. As such, the explosibility parameters can be expected to be similar to those of a turbulent gas deflagration. While the P_{\max} is not notably different, the K_G has been reported to be greater than 500 bar.m/sec (Britton and Chippett, 1989), well above any expected dust values.

The proposed phenomenon was first observed with anthraquinone. Test results, shown in the Figure 17 for anthraquinone, show a K_{St} increase by more than 220% when comparing 20-L sphere to 1-m³ chamber. Anthraquinone has a reported flash point temperature of 185°C, a melting point at 286°C and a boiling point at 380°C, well below the maximum temperature produced by the pyrotechnical igniters as measured by Scheid et al. (2013). It is reasonable that the chemical igniter is able to induced sufficient sublimation and vapor formation to change the combustion scenario from dust to hybrid.

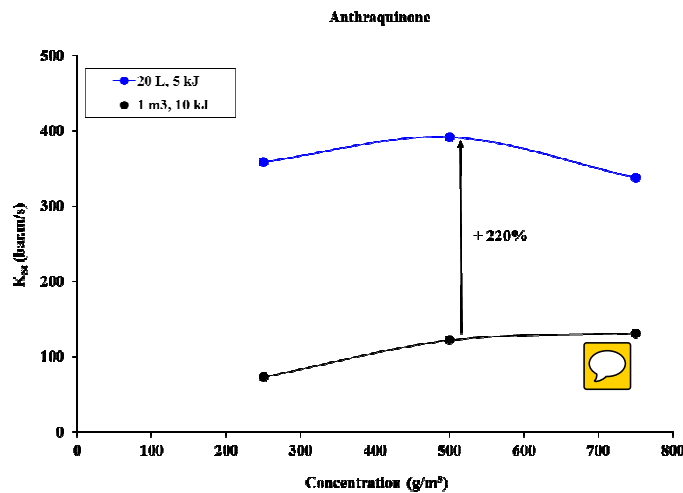



Figure 17: K_{St} values in a 20-L sphere and 1-m³ vessel for anthraquinone

The  sample was a microcapsule filled with oil. It is believed that the ignition process was sufficient to fracture the microcapsule and vaporize some of the oil. The test results in Figure 18 indicate a K_{St} increase of 40% at 500g/m³ when comparing 20-L sphere data to 1-m³ chamber data. It is likely that if a 10-kJ pyrotechnical igniter have been used in the 20-L sphere, the K_{St} might have been even higher. Results with 1-kJ in the 20-L sphere gave a slight but significant reduction showing the importance of ignition energy.

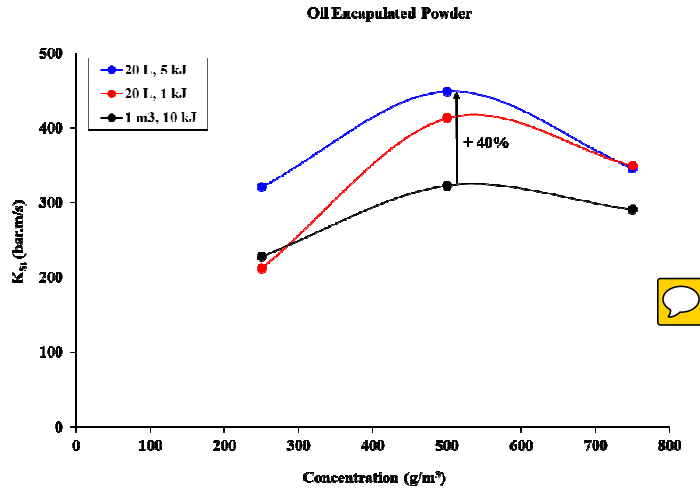


Figure 18: K_{St} values in a 20-L sphere and 1-m³ vessel for an oil encapsulated powder. The tested sample was a wax coated powder. The wax was reported to be low melting. It is believed that the temperature rise from the 5-kJ igniter was sufficient to vaporize the wax coating, resulting in hybrid conditions. In this case, the K_{St} in the 20-L sphere was increased by more than 130% when using a 5-kJ igniter (Figure 19) at 250 g/m³. This sample, tested by another laboratory in a 20-L sphere, exhibited $K_{St} > 400$ bar.m/s as well. These high K_{St} values found in the 20-L sphere suggest a dust hazard that cannot be protected effectively by either venting or suppression.

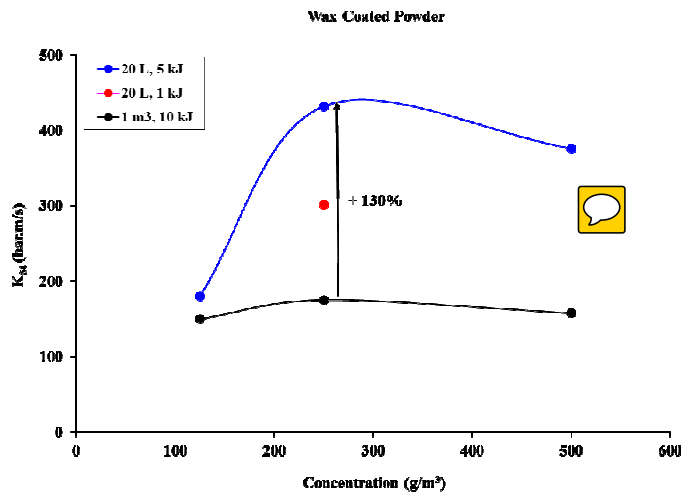


Figure 19: K_{St} values in a 20-L sphere and 1-m³ vessel for wax coated powder

The fourth tested sample was a pigment. The K_{St} in the 20-L sphere was increased by 60% at 250 g/m³ (Figure 20).

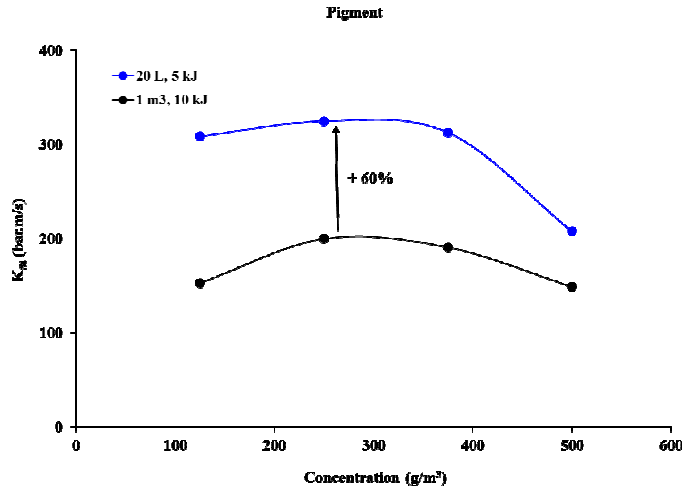


Figure 20: K_{St} values in a 20-L sphere and 1-m³ vessel for a pigment

Figure 21 summarizes the tests done with these four dusts in the 20-L sphere and 1-m³ chamber and show the maximum K_{St} (or K_{max}).

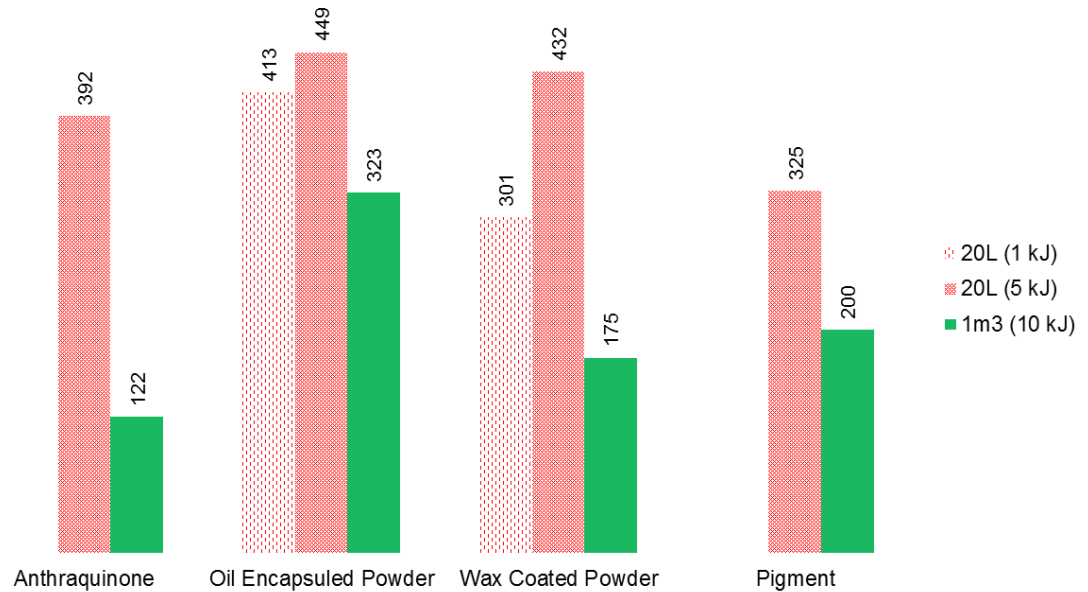


Figure 21: Evolution of K_{St} values for the four tested samples

5. Discussion

It is proposed that a high energy pyrotechnical igniter, when used in a small enclosure, may be physically altering the dust sample being tested. If this results in the formation of a vapor in addition to the original dust, then the final sample will have all the characteristics of a hybrid, turbulent vapor and dust. We have termed this an igniter induced hybrid.

The altering is such that the sample being tested is not the same and the results may not be representative of ignition of the sample in a process equipment.

Synthetic materials which exhibit low flash points, such as polymers or chemicals, are believed to be able to quickly generate combustible vapors by sublimation (i.e. a physical process) upon the activation of the pyrotechnical igniters.

This first mechanism shown in Figure 22 addressing the combustion of anthraquinone and a pigment, is believed to be comparable to droplet combustion.

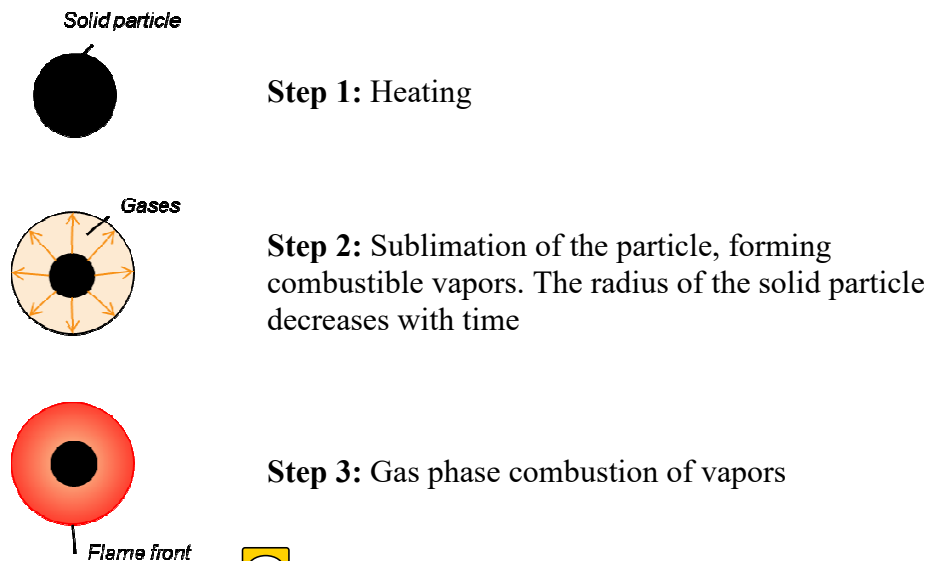


Figure 22. Proposed mechanism (homogeneous material: anthraquinone and pigment)

A powder sufficiently fine and sensitive to elevated temperatures may have high sublimation rates and exhibit this kind of behavior. It is possible that Minimum Auto Ignition Temperature (MAIT) tests can help in identifying such dusts.

On Figure 23, a combustion mechanism is proposed for a heterogeneous material, i.e. a wax coated powder. It is believed that the wax layer vaporized first, creating combustible vapors surrounding the particle.

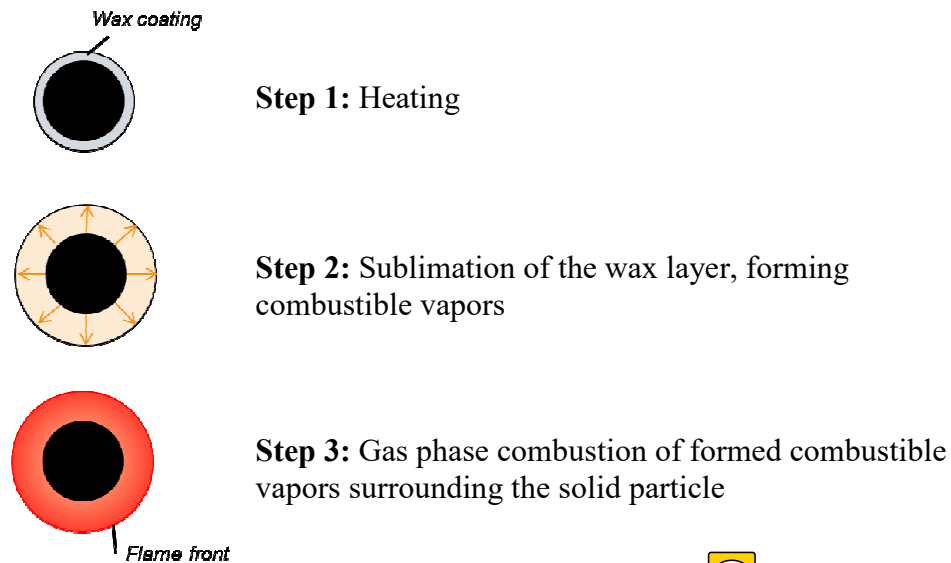


Figure 23. Proposed mechanism (heterogeneous: wax coated powder)

The combustion of the oil-encapsulated powder is illustrated on Figure 24. The oil is released upon heating and break-up of the particles. Liquid droplets are released, probably vaporized and burned around the particle.

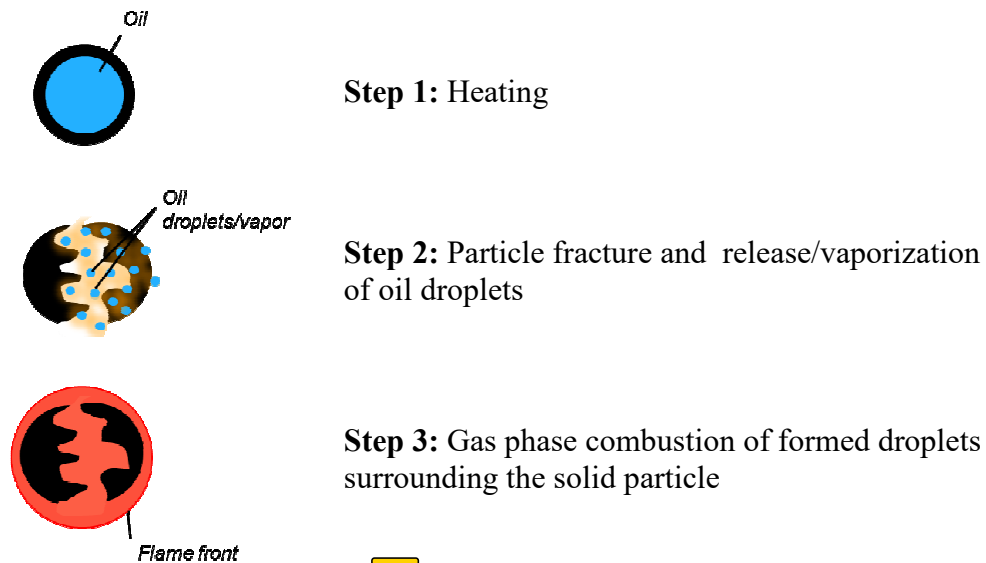


Figure 24. Proposed mechanism (heterogeneous: oil encapsulated powder)

It is interesting to observe that, using the same injection protocol established for dust testing in a 26-L sphere, Britton and Chipplet (1989) obtained K_G values of 510 bar.m/s for methane and 635 bar.m/s for propane. These K_G values are comparable to the K_{St} values presented on Figure 21 for the 20-L sphere. This tends to demonstrate that the experiments carried out in the 20-L sphere with the 4 dust samples described above actually involved turbulent dust-gas mixtures.

It is recommended to carry out additional tests in a 1-m³ chamber when pyrotechnical igniters employed in the 20-L sphere are suspected to alter the physical characteristics of the dust sample.

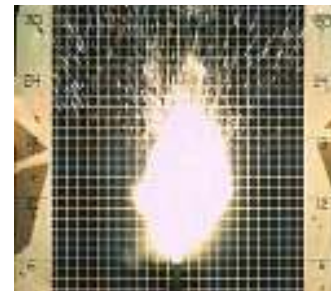
Using less “invasive” ignition sources is another potential solution. For comparison purposes, Figure 25 shows the fireball created by a fuse wire, a gel cap and a 5-kJ pyrotechnical igniter.



Fuse wire



Gel cap



**5 kJ
pyrotechnical igniter**

Figure 25: Visualization of the fireball generated by different ignition sources

6. Conclusions

For the first time, evidence has been provided that pyrotechnical igniters may, in some instances, physically alter the dust being tested in a 20-L sphere.

It is likely that the elevated temperature from the igniter affects the dust prior to flame arrival, and is responsible for the physical changes in the dust that result in a hybrid mixture¹. We proposed to name it an igniter induced hybrid.

This behavior depends on the physical properties of the dust and may be characterized, in some instances, by the flash point or the MAIT of the dust being tested.

In our experiments, K_{St} values obtained in the 20-L sphere were 2 to 4 times greater than in the 1-m³ chamber. Therefore, the results from 20-L sphere testing were no longer representative of a dust deflagration in a real process environment.

In light of the results provided in the present paper, and as a rule of thumb, when a dust exhibit a low flash point, or when its K_{St} in the 20-L sphere is above 300 or 400 bar.m/s, then it is possible that the combustion reaction has been overdriven by the pyrotechnical igniters. Therefore, it is recommended to carry out additional tests in a 1-m³ chamber, which remains the reference vessel for determining dust explosibility parameters. This recommendation maintains consistency with ASTM E1226 standard.

The practical consequences are important, since the significance of this phenomenon is over-interpretation of the hazard, and overdesign of safety measures. Such high reported K_{St} values may lead to impractical explosion protection designs, as well as expensive process or equipment modifications.

As an example, the high K_{St} values (> 400 bar.m/s) found in the 20-L sphere for the wax coated powder suggest a dust hazard that cannot be protected effectively by either venting or suppression.

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¹ In physics, the Observer Effect states that the measurement of some systems cannot be made without affecting the system.

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